

EPA NATIONAL LEAD LABORATORY ACCREDITATION PROGRAM

LABORATORY QUALITY SYSTEM REQUIREMENTS (LQSR)

REVISION 2.0 (August 1, 1996)

The requirements in this document apply to all laboratories participating in the Environmental Protection Agency's (EPA) National Lead Laboratory Accreditation Program (NLLAP). In addition to meeting the Laboratory Quality System Requirements (LQSR), the laboratory must also successfully participate in the Environmental Lead Laboratory Proficiency Testing (ELPAT) Program. The ELPAT Program is a cooperative effort of the American Industrial Hygiene Association (AIHA), researchers at the Centers for Disease Control and Prevention (CDC), the National Institute for Occupational Safety and Health (NIOSH), and the U.S. EPA, Office of Pollution Prevention and Toxics (OPPT).

Concerning any future revisions to the NLLAP Laboratory Quality System Requirements, laboratories currently participating in NLLAP will be given a period of six months from the posting of the revision on the Government Printing Office's Federal Bulletin Board to conform to any new requirements stated in the revision. Note the training requirements stated for analysts and technicians in this revision apply only to laboratory staff which undergo training after the posting of this revision by Federal Register Notice.

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NATIONAL LEAD LABORATORY ACCREDITATION PROGRAM (NLLAP)

LABORATORY QUALITY SYSTEM REQUIREMENTS

INTRODUCTION AND PURPOSE

All requirements of International Organization for Standardization and International Electrochemical Commission (ISO/IEC) Guide 25: 1990 (E) "General requirements for the competency of calibration and testing laboratories" must be followed by all NLLAP recognized laboratories. In addition, the following criteria are required and expand upon the ISO/IEC requirements. This document identifies the minimum requirements for use by accreditation organizations to evaluate laboratories performing environmental testing activities under NLLAP. NLLAP recognizes laboratories that perform quantitative analytical testing of paint chip, dust, and/or soil samples **collected** for lead analysis. NLLAP requires that laboratories employ quality assurance/quality control systems, as described in this document, to monitor for potential sample matrix and environmental interferences as well as laboratory operational deficiencies.

1. ORGANIZATION AND FUNCTION

1.1 Laboratory Types

NLLAP recognizes three types of laboratory operations: fixed-site, mobile and field operation. For the purposes of NLLAP, these types of laboratory operations are defined as follows:

Fixed-Site:	An operation which performs analytical testing from a permanent location associated with improved real estate.
Mobile Facility:	A transportable, self contained facility that can perform analytical testing under controlled environmental conditions.
Field Operation:	A temporary operation, using portable testing technologies, which performs analytical testing on-site, near the sampling location under evaluation.

1.2 Laboratory Entity Accreditation

Organizations desiring accreditation for multiple laboratories shall submit separate applications for each entity. A laboratory entity is defined as a discrete unit based upon unique characteristics including laboratory type, physical facilities, distinct staffing, instrumentation and methods used. Accreditation of the laboratory entity shall be based on its meeting the NLLAP LQSR. Each entity shall be individually accredited. For example, a laboratory meeting the definition of a mobile facility shall only be accredited as a mobile facility.

1.3 Lead Analysis Capability

The laboratory for which the organization is requesting accreditation shall possess a lead analysis capability constituting an identifiable part of the overall laboratory operation.

2. QUALITY SYSTEM

A laboratory's quality system shall include all quality assurance (QA) policies and quality control (QC) procedures. These procedures shall be contained in the quality systems manual and related quality system documentation described in Section 2.2, to help ensure and document the quality of the analytical data.

All items in the laboratory's quality system shall be available for on-site inspection or audit by EPA NLLAP personnel and by accreditation organizations participating in NLLAP.

2.1 Quality System Establishment

The laboratory shall establish and maintain a quality system.

- a. The elements of this system shall be documented.
- b. The quality documentation shall be available for use by laboratory personnel.
- c. The laboratory management shall ensure that these policies and objectives are documented in a quality assurance manual and communicated to, understood, and implemented by laboratory personnel.
- d. The quality assurance manual shall be updated and maintained under the responsibility of the quality manager (or however named).

2.2 Quality System Documents

The quality systems manual (QSM) and related quality systems documentation shall state the laboratory's policies and operational procedures established in order to meet the NLLAP LQSR.

The current version of the laboratory QSM shall be made available to the accreditation organization upon initial application and with each subsequent reaccreditation application. The QSM and related documents shall reflect the implemented quality system in operation at the laboratory and include an organizational chart identifying key personnel, responsibilities, lines of authority, and interrelationships of staff. The QSM and related quality system documents shall also include QC requirements for methods used by the laboratory.

The QSM and related quality systems documents shall include or address at least the following elements:

- # Title Page
- # Table of Contents
- # Organization and Responsibility
- # Quality Assurance Objectives and Policies
- # Personnel Qualifications and Training
- # Sampling Procedures (when applicable)
- # Sample Receipt/Chain of Custody Procedures
- # Major Instrumentation and Equipment
- # Reagents and Standards
- # Equipment Calibration and Maintenance Procedures
- # Sample Preparation and Homogenization Methods
- # Analytical/Testing Methods
- # Data Reduction, Validation and Reporting
- # Internal Quality Control Procedures
- # Performance and System Audits and Reporting
- # Corrective Action
- # Documentation and Record Keeping
- # Sample Retention and Disposal
- # Procedures for Dealing with Client Complaints

The QSM shall be updated whenever necessary and reviewed and approved by management at least annually. The QSM shall be accessible to all laboratory personnel.

3. FACILITIES

Any laboratory shall have, as appropriate, the space, equipment, instruments, ventilation, utility services, storage space, safety equipment, and documentation and references necessary to accomplish the analyses for lead concentrations in the matrices of concern (see 29 Code of Federal Regulations [CFR] 1910.1450).

3.1 Sample Receipt and Storage

Appropriate area and equipment shall be provided for sample receipt, storage and processing.

3.2 Environmental Control

Temperature, humidity, ventilation, dust and vibration shall be controlled to meet instrument and/or sample analysis requirements.

3.3 Mobile and Field Operation Laboratory Locations

A historical record of mobile laboratory and field operation locations shall be maintained for at least ten years and include a specific description of where the analytical work was performed.

4. PERSONNEL QUALIFICATIONS, RESPONSIBILITIES AND TRAINING

The laboratory personnel involved in NLLAP analyses must be educated, trained, experienced and skilled to conduct the various functions performed by the laboratory. The criteria and training requirements for laboratory personnel shall be clearly defined, documented, and maintained on file. The documentation shall include a description of the training program content, the duration of the training, qualifications of the trainer, and evidence that the analyst/technician has successfully performed the number of required test runs (See Section 4.4.1.4).

The laboratory shall have job descriptions for all positions. As a part of a laboratory's quality system documentation, an organizational chart shall be available identifying the functions of key personnel, their responsibilities, lines of supervision, and authority. Laboratory management shall be responsible for all personnel employed by the laboratory including those assigned to mobile or field operation laboratories.

All NLLAP laboratories must identify a responsible laboratory official who authorizes the release of the final data report on behalf of the laboratory.

Laboratory personnel shall consist at a minimum, of a technical manager and a qualified person (See Section 9.1) not directly involved with the analysis of the sample set, to review and concur on the data for use in the final report. Although the position of quality assurance manager (however named) is also a requirement, the technical manager may also function as the quality manager. The quality manager function can also be contracted out. In addition, the following specific personnel requirements shall apply:

4.1 Technical Manager

4.1.1 Qualifications

The individual who functions as the technical manager (however named) of the lead analysis laboratory shall possess a college degree in chemistry or a related science. The technical manager shall have at least three years non-academic analytical chemistry laboratory experience, of which at least two years shall be metals analysis experience.

Note NLLAP requires the technical manager function to be held by a laboratory employee and not contracted out.

4.1.2 Responsibilities

The technical manager shall be responsible for all technical operations and shall be available during at least 50% of the laboratory operating hours to address technical issues for laboratory staff and customers concerning NLLAP related analyses.

The technical manager shall ensure and document that personnel with appropriate educational and/or technical background perform all analyses for which the laboratory is accredited.

The technical manager shall ensure that adequate supervision is provided for all laboratory technical personnel.

4.2 Quality Manager

4.2.1 Qualifications

The individual who functions as the quality manager (or however named) of the lead analysis laboratory shall possess a college degree in a basic or applied science and have training in statistics and at least one year of non-academic analytical chemistry experience. Alternatively, the individual shall have training in statistics and four years of non-academic analytical chemistry experience. The quality manager shall be knowledgeable of the quality system in effect at the laboratory and the analytical methodologies utilized.

4.2.2 Responsibilities

The responsibilities of the quality manager shall include the implementation and oversight of the quality system, the implementation of new quality assurance and control practices, periodic audits of the quality system in place, periodic review of final data reports, the documentation of laboratory quality system deficiencies and the implementation of corrective actions at the laboratory.

4.3 Laboratory Supervisors

This individual shall have a college degree in chemistry or related field with a minimum of one year of non-academic experience in metals analysis. Successful training in specific metals methods including sample preparation techniques, digestion procedures, and operation of instruments used in the laboratory shall be verified and documented using reference materials of the matrices of concern. Proficiency testing results shall be documented.

Individuals without a degree in chemistry or a related field can be recognized as laboratory supervisors as long as they meet the training and proficiency testing requirements stated in the previous paragraph and have demonstrated to be proficient in metals analyses over a period of at least three years. During this time, these staff shall have performed analysis of metals using the same technologies used for the analysis of NLLAP-related lead samples. This demonstration of relevant proficiency shall be documented.

4.4 Analysts/Technicians

For some analytical technologies it may not be possible to isolate sample preparation techniques from instrumental analyses. In such cases, the training requirements shall be based upon the minimum requirements stated for analysts and technicians.

4.4.1 Minimum Qualifications and Training for Analyst/Technician Personnel

4.4.1.1 NLLAP defines an analyst as an individual who performs sample analyses and possesses a bachelor's degree in chemistry or a related science. An individual performing sample analyses that does not have a degree in chemistry or a related science is defined as a technician.

Analysts and technicians shall have a minimum of 30 calendar days of hands on experience conducting analyses in an inorganic/metals laboratory before initiation of work on NLLAP related samples.

4.4.1.2 Analysts shall complete an external and/or internal training program for lead or applicable metals analysis prior to performing analyses on NLLAP samples. Courses on sample preparation and instrumental analysis may be taken separately or combined. The criteria and training requirements for laboratory personnel shall be clearly defined, documented and maintained on file. A description of the training program content, the duration of the training, qualifications of the trainer, and objective evidence that the analyst has successfully analyzed unknown reference samples of the matrices of concern within the specified acceptance criteria must be maintained by the laboratory.

4.4.1.3 The analyst/technician trainee shall become familiar with the SOPs in use in the laboratory and with the instrument and equipment operation manuals.

4.4.1.4 The analyst/technician trainee shall complete a minimum of four independent test runs of sample preparation and/or instrumental analysis. Independent runs are defined as

analytical runs consisting of at least five samples, one of which is a certified reference material or proficiency testing material, separated by a period of time sufficient to evaluate the performance of any previous independent run. For sample preparation training, the recoveries of the associated reference materials or proficiency training samples for each run must be within $\pm 20\%$ of the certified value, 75% of the time. For instrumental analysis training, the recoveries of the associated reference materials or proficiency training samples for each run must be within $\pm 10\%$ of the certified value, 75% of the time.

The reference/proficiency test samples utilized shall: 1) be similar to matrices the analyst will encounter during routine sample analysis, 2) cover the sample mass range for which the analytical SOP has been designed and 3) cover the lead (Pb) concentration for which the analytical SOP has been designed. In cases where there are several matrices of potential concern, four independent runs will not be sufficient to provide adequate demonstration of performance.

- 4.4.1.5 Analyst/Technicians involved in lead analyses shall periodically demonstrate their ability to adequately analyze samples for lead based on standard reference materials (SRMs) or certified reference materials. This demonstration shall be done at a minimum of every six months and can be a part of the analysis of proficiency testing materials or quality control samples associated with routine sample runs.

4.4.2 Additional Requirements for Mobile and Field Operation Personnel

- 4.4.2.1 All mobile and field operation laboratory personnel involved in the designation of sampling areas as a part of a lead-based paint risk assessment in target housing and/or child occupied facilities shall be certified by EPA or an authorized state or tribal program as a risk assessor as pursuant to Section 402 of the Toxic Substance Control Act (TSCA).
- 4.4.2.2 All mobile and field operations personnel shall have the capability to communicate with their supervisor or technical manager while on site at a field job location.
- 4.4.2.3 All mobile or field operation technicians shall be accompanied by a qualified supervisor for their initial two NLLAP-related job sites.

5. REAGENTS AND STANDARDS

Requirements for reagents and standards shall be specified in the documented quality manual.

5.1 Reagent Grades Used

Reagents and standards shall be at least American Chemical Society (ACS) reagent grade or of the quality specified by the analytical methods in use by the laboratory.

5.2 Reagents and Standards Tracking

Purchased reagents and standards shall be inspected, dated and initialed upon receipt. The concentrations of standards shall be certified as National Institute of Standards and Technology (NIST) traceable or verified against NIST standards (if available). Standards shall have an expiration date assigned. Reagents and standards shall not be used beyond assigned expiration dates nor used if damaged or contaminated or suspected to be damaged or contaminated.

The laboratory shall maintain documentation on the manufacturer's statement of purity, of the origin and traceability of all standards and reagents.

5.3 Documentation of Reagent and Calibration Solution Preparation

Documentation of standard and solution preparations shall include the date of preparation, concentration and/or purity of parent material concentration, assigned expiration date and preparer's initials. All prepared reagents and standards must be uniquely identified and the contents shall be clearly identified.

6. SAMPLE ACCEPTANCE, DOCUMENTATION AND TRACKING PROCEDURES

The laboratory shall have a written standard operating procedure (SOP) for sample receiving and tracking. This procedure shall include a laboratory identification system that uniquely identifies each sample and/or batch of samples received by the laboratory or analyzed by a field operation laboratory.

6.1 Sample Acceptance Policy

The laboratory shall have a written sample acceptance policy that clearly outlines the circumstances under which samples will be accepted. Data from any samples which do not meet the following criteria must be clearly flagged defining the nature and substance of the variation. This sample acceptance policy shall be made available to sample collecting personnel and shall include, but is not limited to, the following areas of concern:

- a. Proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, preservation type (where relevant), sample matrix and any special remarks concerning the sample;
- b. Proper sample labeling that includes unique identification.
- c. Use of appropriate sample containers;
- d. Adequate sample mass in order to perform the analysis.

6.2 Sample Receipt Logs

The laboratory shall utilize a permanent chronological record, such as a log book or electronic record, to document receipt of all samples. The following information must be recorded in the laboratory log:

- a. Date of laboratory receipt of sample;
- b. Sample collection date (if known);
- c. Unique laboratory ID code (See Section 6.3);
- d. Field ID code supplied by sample submitter;
- e. Sample matrix;

- f. Requested analyses, including approved method number, if applicable;
- g. Signature or initials of sample logger (where applicable); for electronic sample logging systems, the identity of the logging operator;
- h. Comments resulting from inspection for sample acceptance rejection

All documentation, such as memos or transmittal forms, that is transmitted to the laboratory by the sample transmitter shall be retained.

6.2.1 Documentation on Questionable Samples

Where there is any doubt as to the sample's suitability for analysis testing, where the sample does not conform to the description provided, or where the analysis required is not fully specified, the laboratory shall consult the client for further instruction before proceeding. The laboratory shall establish whether the sample has received all necessary preparation, or whether the client requires preparation to be undertaken or arranged by the laboratory. If the sample does not meet the sample acceptance criteria, the laboratory shall:

- 6.2.1.1 Retain correspondence and/or records of conversations concerning the final disposition of rejected samples or fully document any decision to proceed with the analysis of compromised samples.

The condition of these samples shall, at a minimum, be noted on the chain of custody or transmittal form and laboratory receipt documents.

- 6.2.1.2 The analysis data shall be appropriately "qualified" on the final report.

6.2.2 Sample Custody Procedures

The use of legal chain of custody (COC) protocols is strongly recommended and may be required by some state or federal programs. The chain of custody records shall establish an intact, continuous record of the physical possession, storage and disposal of collected samples.

6.3 Sample Tracking

The laboratory shall have a documented system for uniquely identifying the items to be analyzed, to ensure that there can be no confusion regarding the identity of such items at any time. This system shall include identification of all samples, subsamples, and subsequent extracts and/or digestates. The laboratory shall assign a unique identification (ID) code to each sample container received in the laboratory. Multiple aliquots of a sample that have been received for different analytical tests must be assigned a different ID code (such as a prefix or suffix).

7. EQUIPMENT AND INSTRUMENTATION

All equipment used to prepare samples for instrumental analysis shall be maintained and calibrated in accordance with manufacturer standards, as well as any specifications stated in the analytical methods. Standards used for calibration shall be traceable to NIST standards (when available).

Each instrument used in the analysis of lead shall have SOPs for calibration/verification and be readily available to the analysts. Calibration/verification and maintenance procedures shall be specified and recorded for support equipment.

7.1 Instrument Maintenance Records

Records shall be maintained for each major instrument, including records of in-house preventive maintenance and service. The frequency of calibration/verification for each instrument shall be documented. Descriptions of the problem or service, dates and types of repair, organization and person performing repair, and contact phone number shall be recorded. Instruments which are out of calibration or defective shall be taken out of service until repaired and demonstrated to be functioning within documented acceptance limits. The record shall identify the instrument by make, model number, serial number, and when available, the date placed in service.

7.2 Instrument Performance Quality Checks/Calibration

Instruments shall be subjected to performance checks prior to use. Such checks may include evaluation of instrument sensitivity, noise levels, and absorbance/emission levels versus historical values. Acceptance criteria shall be stated (See Section 10). Instruments which are amenable to daily routine calibration shall be calibrated on at least a daily basis before analyzing samples.

Section 7.2.1 below provides general instrument calibration requirements. Sections 7.2.1.1 through 7.2.1.4 provide specific calibration requirements for atomic absorption spectroscopy (AAS) and inductively-coupled plasma (ICP) methodologies.

7.2.1 Instrument Calibration General Requirements

- a. All calibration curves shall be dated and labeled with applicable method, instrument identification, analysis date, analyte concentrations, and instrument response.
- b. When used, the axes of the calibration curve shall be labeled. For electronic data processing systems that automatically compute the calibration curve, the equation for the curve and the

correlation coefficient must be recorded. The equation for the line and the correlation coefficient shall also be recorded when the calibration curve is prepared manually.

- c. A criterion for the acceptance of a calibration curve, for example, an acceptable correlation coefficient, shall be established and documented.
- d. When available, all initial calibrations shall be verified with standards obtained from a second or different source. These verification standards shall be analyzed with each initial calibration.
- e. The sample results shall be bracketed by calibration standards under all circumstances.

7.2.1.1 Initial Calibration

A minimum of three calibration standards, which bracket the sample concentrations, and an initial calibration blank (ICB) shall be analyzed to construct a calibration curve on a daily basis before the analysis of samples, as appropriate. Acceptance criteria shall be stated. New curves shall be prepared whenever an out of control condition is indicated and/or after new reagents are prepared. For those technologies and software packages requiring fewer standards, such as ICP, following the operations manual and the SOP is acceptable. Additional standards shall be required in instrumental methodologies where the curve is non-linear.

Linearity shall be confirmed by the calibration standards over the concentration range of interest. Freedom of known interferences that can be checked using interference check standard(s) shall be demonstrated for each calibration period. Acceptance criteria shall be stated (See Section 10).

7.2.1.2 Independent Calibration Verification

An independent calibration verification (ICV) standard shall be analyzed at a minimum frequency of once per day prior to the analysis of samples. The source of the ICV standard shall be independent from the instrument calibration samples and be NIST traceable (See Section 10 for performance criteria). Where applicable, the ICV standard shall be at a lead concentration in the range of client specified lead levels of concern or action levels such as regulatory limits.

7.2.1.3 Continuing Calibration Verification

Continuing calibration verification (CCV) standards shall be analyzed in accordance with the analytical SOP. The CCV standard may be prepared from independent reference standards or from the same

standards used to prepare the instrument calibration curve. Acceptance criteria shall be stated (See Section 10).

- a. These standards shall be analyzed at a frequency of one every ten samples or every 12 hours, or according to manufacturer's recommendations, whichever is most frequent.
- b. The concentration of these standards shall be determined by the anticipated or known concentration of the samples and/or method specified levels. At least one standard shall be at a low level concentration. To the extent possible, the samples in each interval should be bracketed with standard concentrations closely representing the lower and upper range of reported sample concentrations. If this is not possible, the standard calibration checks should vary in concentration throughout the range of reported sample concentrations. If this is not possible, the standard calibration check should vary in concentration throughout the range of the data being acquired.
- c. A new curve shall be run if two back-to-back runs of one continuing calibration check is outside acceptable limits. When the continuing calibration check limit is exceeded high (i.e., high bias), and there are non-detects for the corresponding analyte in all environmental samples associated with the continuing calibration check, then those non-detects may be reported, otherwise, the samples affected by the unacceptable check shall be reanalyzed after a new calibration curve has been established, evaluated, and accepted. Additional sample analysis cannot occur until a new calibration curve is established and verified.

7.2.1.4 Continuing Calibration Blank (CCB)

Continuing Calibration Blank (CCB) standards shall be analyzed in accordance with the analytical SOP.

Note: The method blank can also serve as ICB and/or CCB.

8. SAMPLE PREPARATION AND ANALYSIS METHODOLOGY

8.1 Standard Operating Procedures

Method SOPs shall include information addressing the following areas, as appropriate:

- # method detection limit
- # scope and application
- # summary of the method
- # definitions
- # applicable matrix or matrices
- # applicable lead concentration range
- # applicable sample mass range
- # method performance (accuracy and precision)
- # interferences
- # safety considerations
- # reagents and standards
- # equipment and supplies
- # sample collection (where applicable)
- # sample preservation and storage (where applicable)
- # sample preparation (grinding, homogenization, and subsampling)
- # instrument calibration/verification
- # quality control procedures
- # detailed step-by-step procedures
- # calculations
- # data acceptance criteria
- # corrective actions for out-of-control data
- # contingencies for handling out of control data
- # references

Laboratory SOPs which do not initially address all of the areas stated above may be amended through attachments in order to meet this requirement or reference quality system documents as appropriate. All operating procedures shall be available to each analyst at the testing work area.

8.1.1 Quality Control

The laboratory shall have quality control (QC) procedures stated in their quality system documents including their quality systems manual and/or in each method SOP addressing, as appropriate:

- # Duplicate or "Side-by-Side" Field Sample Analyses
- # Spiked and Blank Sample Analyses
- # Blind Samples
- # Split/Spiked Field Sample Analyses
- # Control Charts or Equivalent
- # Calibration Standards
- # Laboratory Control Samples
- # Internal Standards

8.1.2 SOP Review/Revision

The laboratory shall outline in its QSM or document control SOP, the process utilized in the adoption and revision of analytical procedures employed by the laboratory. This document shall address when and how the laboratory SOPs manual(s) are reviewed, identify the sign off authority, and state that the SOPs are reviewed at least annually and/or revised as needed.

8.2 Acceptable Methodology

- a. Methods shall not be used for sample analysis until competency for analysis of each particular matrix has been demonstrated by the laboratory. Competency shall be demonstrated over the lead concentration and sample mass ranges stated by the method. These methods shall be available to all analysts in the form of written SOPs and dated and approved by the appropriate authority. Laboratory method demonstrations shall be documented.
- b. Where sample preparation and analysis methods are not specified by regulatory programs, the laboratory shall, whenever possible, use procedures published by federal agencies such as EPA or NIOSH, state agencies, or nationally or internationally recognized technical authorities such as ASTM (American Society for Testing and Materials). Methods under consideration for analytical testing shall demonstrate a quantitation limit equal to or less than 20% of the lowest relevant action level or regulatory limit of interest. Laboratory SOPS for sample analysis may require additional QC procedures to those stated in the published methods in order to meet NLLAP performance requirements.

Testing method guidance for paint chip, dust and/or soil samples may be obtained from the Department of Housing and Urban Development (HUD) "Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing" (July 1995).

- c. Alternative or modified analytical methods may be used by a laboratory if they have been validated by the laboratory meeting the minimum performance requirements specified in Section 10 of this document. The method validation must be documented by the laboratory.

Possible method validation procedures:

- # U.S. EPA Office of Solid Waste "Guidance for Method Development and Method Validation for the RCRA Program, June 1995."
- # Guidance on Evaluation, Resolution, and Documentation of Analytical Problems Associated with Compliance Monitoring, US EPA Office of Water, EPA 821-B-93-001, June 1993.
- # Guidelines for Collaborative Study Procedure to Validate Characteristics of a Method of Analysis, J. Assoc. Off. Chem., Vol. 72, No.4, 1989, p. 694
- # AOAC® Peer - Verified Methods Program, Nov. 1993.

8.2.1 Method Detection Limits

Method detection limits (MDLs) shall be established and statistically verified at least annually for each method and matrix of concern. For methods with stated MDLs, demonstration of ability to achieve such MDLs is required and shall be documented. MDLs shall be determined using procedures published or recognized by federal agencies such as EPA, or NIOSH, or nationally or internationally acknowledged technical authorities.

8.2.2 Accuracy and Precision

Accuracy and precision shall be determined for each analytical methodology utilized. The method evaluation results shall be documented and kept on file. Acceptable method performance criteria for bias and precision can be found in Section 10, Table 1 of this document.

8.3 Sample Aliquots

Where subsampling (obtaining sample aliquots from a submitted sample) is carried out as part of the analytical method, the laboratory shall use documented procedures and appropriate statistical techniques to obtain representative subsamples.

9. DATA REDUCTION, VALIDATION AND REPORTING

9.1 Data Reduction and Review Process

The data reduction and review process shall include, but not necessarily be limited to: comparison of quality control data against established acceptance limits, computation verification, transcription of data, and adherence to the procedures established in the laboratory SOPs. The review process shall be documented. All final test reports shall undergo the documented data review process before release to the client. All data reviews shall be conducted and signed by a qualified person not directly involved in the physical preparation and/or analysis of the samples in question. Qualified persons maybe defined as technicians, analysts, supervisors, technical managers and/or quality managers as described in Section 4.0.

9.2 Data Correction Process

Any corrections made to data shall be documented with a single strike out line and the analyst's initials and date. All handwritten data shall be recorded using indelible ink. No correction fluid shall be used on original laboratory data records.

9.3 Laboratory Test Reports

9.3.1 Final Test Reports

Final test reports shall contain, at a minimum the information outlined below. The client may request additional information to be included in the report. Along with the final test report, the laboratory shall maintain a sample case file or be able to assimilate the sample case information described in Section 11 of this document for a period of at least ten years after the final test report is issued.

Each report shall include at least the following information:

- a. title, e.g., "Test Report", or "Test Certificate", "Certificate of Results" or "Laboratory Results;"
- b. name and address of laboratory, location where the analysis was carried out, if different from the address of the laboratory, and name and phone number of contact person for questions;

- c. unique identification of the certificate or report (such as serial number) and of each page, and the total number of pages;
- d. name and address of client, where appropriate, and project name if applicable;
- e. characterization, condition and clear identification of the analyzed samples;
- f. date of receipt of the sample and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 48 hours;
- g. identification of the analytical method used, or unambiguous description of any non-standard method used;
- h. reference to sampling procedure, where relevant;
- i. any deviations from, additions to or exclusions from the analytical method, and any other information relevant to a specific analytical method such as environmental conditions including the use of relevant data qualifiers;
- j. analytical test results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified; identification of whether data is calculated on a dry weight or wet weight basis; and identification of the reporting units such as µg/l or mg/kg;
- k. synopsis of analytical quality control results focusing on any quality control sample measurements which did not meet the method specified requirements;
- l. a signature and title, or an equivalent identification of the person(s) accepting responsibility on behalf of the laboratory for the content of the certificate or report (however produced), and date of issue;
- m. where relevant, a statement to the effect that the results relate only to the items analyzed.

9.3.2 Corrections

If corrections or additions to a test report are made, they shall be documented and the report reissued as an amended report.

9.3.3 Reporting Values Below the Quantitation Limit

Report of zero concentration is not permitted. Laboratories shall establish a method of limiting the lower reported values to a positive finite lead level that is appropriate for the technology being utilized. Measured lead levels below this positive finite value shall be reported as “less than” (“<”) this positive finite value.

The quantitation limit shall be reported as “less than” (“<”) a value no greater than 10 times the method detection limit as determined in Section 8.

10. QUALITY ASSURANCE AND QUALITY CONTROL

The laboratory quality control program shall include the continual evaluation of its performance (system process control) for each matrix analyzed and shall include determinations of bias and precision, and document the successful participation in the ELPAT Program for each matrix analyzed. One possible method used for laboratory system process control is the use of control charts to monitor the performance of a specific QC sample. Control charts shall specify warning and action limits for acceptance or rejection of QC data. In the absence of a statistically sufficient data base to determine the necessary frequency for QC samples, the laboratory must default to the use of frequencies for QC samples stated in Table 1.

Note: Table 1 contains the required minimum performance criteria and QC sample frequency for analytical SOPs for which no QC sample frequency determinations are available. Frequency determinations are normally based on the use of system process control data produced by the laboratory for the specific method utilized.

10.1 Routine Quality Control Requirements

Matrix spike analyses shall be performed using field samples whenever possible in order to monitor for potential field sample matrix interferences. For field samples too small and difficult to homogenize and split in order to obtain samples for matrix spike evaluation or replicate analyses, the laboratory shall select alternative QC options. One of these options is the analysis of duplicate laboratory control samples for each batch in order to monitor laboratory performance.

10.1.1 Bias Determination

10.1.1.1 Laboratory Control (External Reference) Samples

Laboratory control samples (LCS) used to determine the degree of bias shall be analyzed with a minimum frequency of one per twenty field samples or batch, whichever is most frequent. A batch is defined as a set of samples which are processed (in example, homogenized, digested and analyzed) in one operation.

Note: LCS sources include but are not limited to: proficiency testing samples from the Environmental Lead Proficiency Testing Program (ELPAT), commercially available certified reference samples, or samples prepared from sources different than calibration standards and are of known concentration determined using definitive methods. The use of NIST Standard Reference Materials (SRMs) as a LCS is discouraged when an alternate LCS source is available. The lead concentration of the LCS shall

be near (preferably no greater than) the level of concern or action level and whenever possible shall not require extensive pretreatment dilution or concentration prior to analysis. All external reference sample materials shall be NIST traceable.

10.1.1.2 Matrix Spike (Split/Spiked) Field Samples

In an effort to evaluate potential matrix interference, a matrix spike sample shall be analyzed with a minimum frequency of 5% of the samples for each matrix type per batch of samples. If there are fewer than twenty samples in a batch, at least one matrix spike for each matrix per batch shall be analyzed.

Matrix spike samples shall be prepared using a split field sample (before any digestion process). When possible, the split sample chosen shall be one identified with the lowest concentration of lead detected and the level of lead spiked shall be enough to result in a final lead concentration of the prepared sample of twice the sample's observed native lead concentration, or five times the method detection limit, whichever is greatest.

10.1.1.3 Method Blanks

A method blank containing all reagents and subject to all preparation steps shall be processed and analyzed along with the samples. Method blanks shall be analyzed with a minimum frequency of 5% of the samples for each matrix per batch of samples. If there are fewer than twenty samples in a batch, at least one method blank for each matrix per batch shall be analyzed. Method blanks shall not be used to correct sample results.

10.1.2 Precision Determination

A split field sample (the initial sample being split into two fractions before digestion and analysis) for precision determination shall be analyzed with a minimum frequency of 5% of the samples for each matrix type per batch of samples. If there are fewer than twenty samples in a batch, at least one test sample for each matrix per batch shall be analyzed. For analyses where there is not sufficient enough field sample for splitting or the analytical technology does not allow for split samples, the laboratory shall use alternative QC procedures in an effort to monitor the laboratory's precision of analysis.

10.2 Control Charts

Control charts or a quality control data base shall be used to record quality control data and track laboratory performances with respect to the associated acceptance limits for each matrix. The performance tracking of critical QC samples shall be done as close as possible on a real time basis.

10.3 Contamination Control

10.3.1 Laboratory Dust Wipe Checks

For fixed-site and mobile laboratory facilities, wipe sampling and analysis of sample preparation and analysis area surfaces shall be conducted at least quarterly to determine surface concentration levels of lead (Pb). Sample preparation and analysis is not to proceed until surface contamination is within the specified maximum allowable concentration of **50 μ g/ft²**. For field operation laboratories, appropriate contamination control blank samples shall be run in order to monitor potential lead contamination.

10.3.2 Labware Cleaning

Cleaning procedures for labware shall be specified by the laboratory in a written SOP. The procedure shall include, where applicable, a periodic monitoring of lead concentrations in cleaning baths, the monitoring of glassware contamination during the analysis of reagent or other blanks, and periodic monitoring of disposable labware.

10.3.3 Sampling Media

Where the laboratory is responsible for supplying sampling media, the media shall be evaluated as appropriate for lead contamination. The evaluation SOP shall be documented and results of evaluation recorded.

TABLE 1 SUMMARY OF QC SAMPLE PERFORMANCE REQUIREMENTS

All instrument performance checks shall be performed using standard materials of the same matrix as the samples being measured. These standard materials shall be traceable to NIST standards (when available). In the absence of sufficient data for statistical determination of adequate QC sample frequency, the QC samples and minimum frequencies shown below shall be performed. Required acceptance limits are also stated.

QC SAMPLE	FREQUENCY	ACCEPTANCE LIMITS
Independent Calibration Verification (ICV)	Once per day after calibration	Within $\pm 10\%$ of known value
Initial Calibration Blank (ICB)	Once per run at the beginning of the run	Absolute value not more than 10% of the regulatory limit or minimum level of concern
Continuing Calibration Verification (CCV)	Beginning & at the end of a sample run as well as every 10 samples or as specified in the SOP	Within $\pm 15\%$ of known value for ICP or FAAS; within $\pm 20\%$ for GFAA.
Interference Check Sample (ICS)	Beginning & end of each run or twice every eight hours	Within 20% of known value
Continuing Calibration Blank (CCB)	After each ICS and CCV	Absolute value not more than 10% of regulatory limit or level of concern
Laboratory Control Sample	One per 20 samples or batch (min. frequency 5%)	Within $\pm 20\%$ of known value
Matrix Spike Sample	One per 20 samples or batch (min. frequency 5%)	Within $\pm 25\%$ of calculated value
Duplicate Field Sample	One per 20 samples or batch (min. frequency 5%)	Within $\pm 25\%$ Relative Percent Difference (RPD)
Method Blank	One per 20 samples or batch (min. frequency 5%)	Absolute value not more than 10% of regulatory limit or level of concern

10.4 Quality Systems Management Review

The quality system adopted to satisfy the requirements of the NLLAP LQSR shall be reviewed at least once a year by the laboratory management to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements.

Such reviews shall be carried out by trained and qualified staff who are, whenever possible, independent of the activities to be audited.

10.5 Corrective Actions

The laboratory shall implement general procedures to be followed to determine when quality control data is out of control:

- a. Identify the individual(s) responsible for assessing each QC data type;
- b. Identify the individual(s) responsible for initiating and/or recommending corrective actions;
- c. Define how the analyst should treat a data set if the associated QC measurements are unacceptable;
- d. Specify how out-of control situations and subsequent corrective actions are to be documented; and
- e. Specify procedures for management (including the QA officer) to review corrective action reports.

If the reported values of QC samples fall outside the applicable acceptance limits, the affected batch of samples shall be reanalyzed whenever possible (See Section 7.2.1.3, c for exceptions). This analysis shall include a new set of QC samples. When possible, sample reanalysis shall initiate with the sample preparation stage.

Laboratories shall document, investigate and take corrective action for all episodes where the QC data shows an out-of-control situation. No data shall be reported until the cause of the problem is determined and corrected, or the laboratory demonstrates the cause was a random event. The laboratory shall keep records of all out-of-control events, the determined cause(s), and corrective action(s) taken. Laboratories shall respond to client quality complaints and maintain records of corrective action.

11. DOCUMENT AND RECORD RETENTION

11.1 General Record Management Requirements

The records for each analyses shall contain sufficient information so that it may be repeated. The record-keeping system must allow historical reconstruction of all laboratory activities that produced the resultant sample analytical data. The history of the sample must be readily understood through the documentation.

- a. All records (including those pertaining to calibration and test equipment), certificates, and reports shall be safely stored, held secure and in confidence to the client. Records shall be available to the accrediting authority and NLLAP representatives.
- b. All records of an organization that are pertinent to a specified project shall be retained for a minimum of ten years. All hardware and software necessary for the historical reconstruction of data must be maintained by the laboratory.
- c. Records that are stored or generated by computers shall have a hard copy or write-protected backup copies.
- d. The laboratory shall establish a document control system which assures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force.
- e. Access to archived information shall be documented with an access log. These records shall be protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.
- f. In the event that a laboratory transfers or goes out of business, the laboratory shall have a plan to ensure that the records are maintained or transferred according to the client's instructions and applicable regulations.
- g. The records shall include the identity of personnel involved in sampling, preparation, calibration, analysis and final reporting.
- h. The record keeping system shall facilitate the retrieval of all working files and archived records for inspection and verification purposes.

- i. Entries in records shall not be obliterated by methods such as erasures, overwritten files, or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction.

11.2 Specific Sample Documentation

A record of all procedures to which a sample is subjected while in the possession of the laboratory shall be maintained in a case file or be able to be generated upon request for a period of ten years. These shall include, but are not limited to, records of:

- a. Sample identification, receipt, acceptance, or rejection and log-in;
- b. Sample storage and tracking including shipping receipts;
- c. Sample preparation records, instrument printouts, and calculations;
- d. Sample analysis logs;
- e. Standard and reagent origin, receipt, preparation, and use;
- f. Equipment and instrument operating conditions;
- g. Calibration criteria, frequency and acceptance criteria;
- h. Data and statistical calculations, review, confirmation, interpretation, assessment, and reporting conventions;
- i. Method performance criteria;
- j. Quality control protocols and assessment;
- k. Records storage and retention; and
- l. Sample disposal, including the date of disposal.

11.2.1 Laboratory Support Activities

In addition to documenting all the above mentioned activities, the following shall be retained:

- a. All original raw data, whether hard copy or electronic, for calibrations, samples, and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- b. A written description or reference to the specific method used which includes a description of the specific steps in the calculation used to translate parametric observations into a reportable analytical value;
- c. Copies of final reports;
- d. Archived standard operating procedures;
- e. Correspondence relating to laboratory activities for a specific project;
- f. All corrective action reports, audits, and audit responses;
- g. Performance evaluation results and raw data; and
- h. Data review and cross checking.

11.3 Computer and Electronic Data

Where computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of data, the laboratory shall ensure that:

Electronic computer records can satisfy the record-keeping requirement without hard copy files if hard copies can be generated as needed. The computer programs shall be validated before they are used and verified on a regular basis through spot checks of computer calculations. Computer file back up procedures are required.

11.4 Administrative Records

The following shall be maintained:

- a) Personnel qualifications, experience, and training records;
- b) Initial and continuing demonstration of proficiency for each technician/analyst;
- c) A log of names, initials, and signatures for all individuals who are responsible for signing or initialing any laboratory record.

11.5 Legal or Evidential Custody Procedures

The chain of custody records shall establish an intact, contiguous record of the physical possession, storage, and disposal of collected samples.

The COC records shall include signatures of all individuals who had access to individual samples.

11.6 Record Retention Period

The policies shall include the manner and duration of record retention. All laboratory records shall be maintained for a period of at least ten years. If the laboratory closes its operation, the laboratory shall notify all clients who have requested analyses under NLLAP in the past ten years of its intent to close.

11.7 System Audits

Final reports of all laboratory systems audits (both internal and external) for operations associated with NLLAP relevant samples shall be kept on file for 10 years.

11.8 Complaints

The laboratory shall have documented policy and procedures for the resolution of complaints received from clients or other parties about the laboratory's activities. A record shall be maintained of all complaints and of the actions taken by the laboratory. Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with policies or procedures, the requirements of the NLLAP LQSR, or the quality of the laboratory's analyses, the laboratory shall ensure that those areas of activity and responsibility involved are promptly reviewed.

12. SAMPLE RETENTION AND DISPOSAL

The sample retention and disposal policies of the laboratory shall be stated.

12.1 Sample Retention

The policies shall include documenting the manner and duration of sample retention.

12.2 Sample Disposal

Laboratories shall comply with all applicable federal, state and local regulations regarding environmental contamination and waste disposal.

APPENDIX I

ACRONYMS AND GLOSSARY OF TERMS ASSOCIATED WITH THE NLLAP

Acronyms

AAS	Atomic Absorption Spectroscopy
ACS	American Chemical Society
AIHA	American Industrial Hygiene Association
ASTM	American Society for Testing and Materials
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CDC	Centers for Disease Control
CMD	Chemical Management Division
COC	Chain of Custody
ELPAT	Environmental Lead Proficiency Analytical Testing (AIHA/NIOSH)
EPA	Environmental Protection Agency
HUD	Housing and Urban Development
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma Atomic Emission Spectrometry
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
ICV	Independent Calibration Verification
ICS	Interference Check Standard
ISO	International Organization for Standardization
ISO-IEC	International Organization for Standardization and International Electrochemical Commission
LCS	Laboratory Control Sample
MDL	Method Detection Limit
MOU	Memorandum of Understanding
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NLLAP	National Lead Laboratory Accreditation Program
OPPT	Office of Pollution Prevention and Toxics
PE	Performance Evaluation
PM	Preventive Maintenance
PT	Proficiency Testing
QA	Quality Assurance
QC	Quality Control
QSM	Quality Systems Manual

RE	Relative Error
RPD	Relative Percent Difference
SAP	Sample Analysis Plan
SOP	Standard Operating Procedure
SRM	Standard Reference Material Produced by NIST
TPB	Technical Programs Branch
TSCA	Toxic Substances Control Act

Glossary

Accreditation:	A formal recognition that an organization (e.g., laboratory) is competent to carry out specific tasks or specific types of analyses. See also <u>Certification</u> .
Accredited laboratory:	A laboratory that has been evaluated and given approval to perform a specified analysis or task, usually for a specific property or analyte and for a specified period of time.
Acceptance limits:	Data quality limits specified by the National Lead Laboratory Accreditation Program for analytical method performance.
Accuracy:	The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. See <u>Precision</u> and <u>Bias</u> .
Aliquot:	See <u>Subsample</u> .
Batch:	A quantity of material produced or processed (in example, homogenized, digested and analyzed) in one operation, considered to be a uniform, discrete unit.
Bias:	The systematic error manifested as a consistent positive or negative deviation from the known true value.
Blind sample:	A subsample submitted for analysis with a composition and identity known to the submitter but unknown to the analyst and used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
Calibrate:	To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the calibration standards should bracket the range of planned measurements. See <u>Calibration curve</u> .
Calibration blank:	See <u>Initial calibration blank</u> .

Calibration-check:	See <u>Calibration verification</u> .
Calibration-check standard:	See <u>Calibration verification</u> .
Calibration curve:	The graphical relationship between the known values for a series of calibration standards and instrument responses.
Calibration drift:	The difference between the instrument response and a reference value after a period of operation without recalibration. See <u>Continuing calibration verification</u> .
Calibration standard:	A substance or reference material used to calibrate an instrument.
Calibration solution:	See <u>Calibration standard</u> .
Calibration verification:	See <u>Initial or continuing calibration verification</u> .
Certification:	The process of testing and evaluation against specifications designed to document, verify, and recognize the competence of a person, organization, or other entity to perform a function or service usually for a specified time. See also <u>Accreditation</u> .
Certified Reference Material:	A reference material that has one or more of its property values established by a technically valid procedure and is accompanied by or traceable to a certificate or other documentation issued by a certifying body. See <u>Certification</u> and <u>Reference material</u> .
Chain of custody (COC):	An unbroken trail of accountability that insures the physical security of samples, data, and records.
Check sample:	An uncontaminated sample matrix spiked with known amounts of analytes, usually from the same source as the calibration standards. It is generally used to establish the stability of the analytical system, but may also be used to assess the performance of all or a portion of the measurement system. See also <u>Quality control sample</u> .

Continuing Calibration
Blank (CCB):

A standard solution which has no analyte and is used to verify blank response and freedom from carryover. The CCB should be analyzed after the CCV and after the Interference Check Standard (ICS).

Continuing Calibration
Verification (CCV):

A standard solution (or set of solutions) used to verify freedom of excessive instrumental drift. The concentration to be near mid-range of linear curve. The CCV should be matrix matched to acid content present in sample digestates. The CCV should be analyzed before and after all sample digestes.

Control chart:

A graph of some measurement plotted over time or sequence of sampling, together with control limit(s) and, usually, a central line and warning limit(s).

Control sample:

See Laboratory control sample.

Corrective action:

Action taken to correct a deficiency noted in a technical systems audit. See Deficiency and Systems audit.

Deficiency:

A failure to fully comply with the requirements of NLLAP usually noted during a technical systems audit. See NLLAP and Systems audit.

ELPAT:

Environmental Lead Proficiency Analytical Testing Program. Successful participation in this proficiency testing program on a quarterly basis is required for all EPA/NLLAP recognized laboratories. ELPAT is administered by AIHA in cooperation with NIOSH and EPA.

Initial calibration
blank (ICB):

A standard solution that contains no analyte and is used for initial calibration and zeroing instrument response. The ICB must be matrix matched to acid content present in sample digestates. The ICB should be measured during calibration and after calibration.

Independent calibration
verification (ICV):

A standard solution (or set of solutions) used to verify calibration standard levels. Concentration of analyte to be near mid-range of linear curve which is made from a stock solution having a different

manufacturer or manufacturer lot identification than the calibration standards. The ICV must be matrix matched to acid content present in sample digestates. The ICV should be measured after calibration and before measuring any sample digestates.

Interference check
standard (ICS):

A standard solution (or set of solutions) used to verify accurate analyte response in the presence of possible interferences from other analytes present in samples. The ICS must be matrix matched to the reagent content present in sample digestates.

Internal quality
control:

The routine activities and checks, such as periodic calibrations, duplicate analyses, and spiked samples, that are included in normal internal procedures to control the accuracy and precision of measurements.

Internal standard:

A standard added to a test portion of a sample in a known amount and carried through the entire demonstration procedure as a reference for calibration and controlling the precision and bias of the applied analytical method.

Laboratory control
sample (LCS):

A matrix-based reference material with an established concentration obtained from a source independent of the instrument calibration and traceable to NIST or other reference materials. The LCS is carried through the entire procedure from digestion through analysis as a field sample. The purpose of the LCS is to evaluate bias of the method.

Laboratory systems
audit:

See Systems audit.

Lot:

A set of samples submitted together for laboratory analysis which can be treated as one or more batches.

Matrix:

The component or substrate which contains the analyte of interest.

Matrix spike:

See Spiked sample.

Method blank:

A mixture of all reagents used for the digestion of paint, soil, or dust matrices but without the matrix. This blank, is carried through all steps

of the analysis starting with the digestion step. This blank evaluates the process for contamination from the laboratory.

Method performance:	A general term used to document the characteristics of a method. These characteristics usually include method detection limits, linearity, precision, accuracy and bias.
Method detection limit (MDL):	The minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, qualitatively or quantitatively measured, and reported to be greater than zero.
Mobile laboratory:	A mobile laboratory is a self-contained, mobile facility that moves under its own power or is conveyed on a trailer, and does not remain at a site for more than two years.
NLLAP:	National Lead Laboratory Accreditation Program. This EPA program recognizes laboratories which have demonstrated they are capable of analyzing paint chip (film), dust and/or soil samples for lead .
NLLAP requirements:	Requirements specified by the EPA National Lead Laboratory Accreditation Program (NLLAP) in order to be accredited for lead analysis in paint chip (film), dust, and/or soil matrices by an EPA-recognized laboratory accreditation organization.
Precision:	The degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms.
Proficiency testing:	A systematic program in which one or more standardized samples is analyzed by one or more laboratories to determine the capability of each participant.
Quality assurance (QA):	An integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a

product or service meets defined standards of quality within a stated level of confidence.

Quality control (QC): The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. The aim is to provide quality that is satisfactory, adequate, dependable, and economical.

Quality manager: The manager of the quality system. The quality manager is independent of the analyst and reports directly to management.

Quantitation Limits: The maximum or minimum levels or quantities of a target analyte that can be quantified to a specified accuracy.

Reference material: A material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or assigning values to materials.

Reference standard: See Calibration standard.

Relative percent difference: A term defined as

$$RPD = \frac{|R_1 - R_2|}{\bar{R}} \times 100$$

where $|R_1 - R_2|$ represents the absolute difference in two values and \bar{R} represents the average of the two values.

Run: A set of consecutive sample measurements.

Sample log: The document where sample identification, condition, etc is noted when samples arrive at the laboratory. The log is part of the sample tracking system. See Sample tracking.

Sample tracking: A system of following a sample from receipt at the laboratory, through sample processing and analysis, and to final reporting. The system includes unique numbering or bar coding labels and the use of a sample log.

Site visit:	An on-site visit to a laboratory for the purpose of conducting a systems audit.
Spiked sample:	A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Spiked samples are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Split samples:	Two or more representative portions taken from a sample or subsample and analyzed by different analysts or laboratories. Split samples are used to replicate the measurement of the variable(s) of interest.
Standard operating procedure (SOP):	A written document that details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.
Standard reference material (SRM):	A certified reference material produced by the U.S. National Institute of Standards and Technology and characterized for absolute content independent of analytical method.
Standardization:	The process of establishing the quantitative relationship between a known mass of target material (e.g., concentration) and the response variable (e.g., the measurement system or instrument response). See <u>Calibrate</u> and <u>Calibration curve</u> .
Stock solution:	A concentrated solution of analyte(s) or reagent(s) prepared and verified by prescribed procedure(s), and used for preparing working standards or standard solutions.
Subsample:	A representative portion of a sample.
Systems audit:	A thorough systematic on-site, qualitative review of facilities, equipment, personnel, training, procedures, record keeping, data

validation, data management, and reporting aspects of a total measurement system.

Validation: The process of substantiating specified performance criteria.